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MECHANICAL PROPERTIES OF MI SiC/SiC COMPOSITES AND THEIR CONSTITUENTS (Preprint)

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An analytical model (pcGINA) was further developed and used to predict the elastic properties of CMC textile reinforced composites. The analytical modeling components were constructed within a unit cell constituent-based geometric model to map the 3D spatial description of the fabric preform. The model implemented a hybrid finite element approach and a virtual work technique.

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Mechanical properties of MI SiC/SiC composites and their constituents

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Abstract

A series of 15 melt infiltrated SiC/SiC composites were fabricated per the standard process arrived at by NASA-GRC (01/01 material). This was done based on the in-situ boron nitride process prior to fiber interface coating and subsequent densification. A series of physical property testing was conducted to determine the mechanical properties, cyclic load response, damage progression and the effect of temperature on elastic properties. Other tests addressed properties that are typically assumed such as through thickness elastic modulus and shear modulus. Additionally, in-situ moduli of constituent materials were experimentally evaluated using nano-indentation techniques at room temperature.

An analytical model (pcGINA) was further developed and used to predict the elastic properties of CMC textile reinforced composites. The analytical modeling component was constructed within a unit cell constituent-based geometric model to map the 3D spatial description of the fabric preform. The model implemented a hybrid finite element approach and a virtual work technique. Moduli data from nano-indentation were utilized to calculate the elastic properties of the CMC composite at room temperature (RT) and data available in literature was used to evaluate elastic properties at 1204 ^{o}C. The model provided good estimates for in-plane normal and shear moduli and Poisson's ratio at room temperature and acceptable estimates for high temperature properties. However, experimental data for out-of-plane compressive modulus, measured at a low stress level, utilizing the stacked-disk method was much lower than calculated values. Micrographic images showed voids within yarns and further experimental evaluation of this method, for the SiC/SiC material as well as a monolithic glass ceramic, showed modulus-value dependence on applied stress, only evident at low stress levels possibly due to surface asperities of stacked disks.

Introduction

Ceramic matrix composites are currently considered for applications in gas turbine engines as well as other high temperature applications. Barriers to successful application of these materials include the lack of appropriate design tools that are necessary to predict the as-manufactured properties and develop design procedures utilizing the full range of 2D and 3D fiber architectures.

One of the corner stones in the development of material models for CMCs is the realization of the multi-component nature of these materials in contrast to typical two or three phase composites (i.e., fiber/matrix or fiber/interphase/matrix). In the SiC/SiC composites under investigation, fibers are coated to increase toughness and to provide environmental protection with one or more layers of boron nitride [1,2]. These fibers are woven or braided into a fabric preform. The matrix is then introduced to the fabric preform in three consecutive densification steps. These include chemical vapor deposition (CVI) of SiC, slurry cast (SC) SiC and a melt infiltrated (MI) silicon metal leaving some entrapped voids. Properties of each of these constituents are dependent on the manufacturing method and the environment limiting the ability to obtain properties of distinctive "stand-alone" phases. Figure 1 shows a micrograph of a 5HS iBN-Sylramic® weave and the various phases of the material. This number of constituents and their interfaces combined with a variable architecture preform outline a formidable task that modelers face in order to predict their behavior.

To overcome these difficulties and to allow the development of robust and efficient analytical tools, a systematic testing methodology is needed to not only determine the suitability of ceramic composites for a specific application but also to aid model development and verification. The effect of temperature and time on each composite constituent needs to be isolated and investigated to develop an understanding of the contribution of each constituent to the overall composite behavior. In this work, efforts towards this goal were carried out utilizing an extensive experimental program and a micromechanics approach known as pcGINA [3].

Experimental program

Materials and Manufacturing

The material chosen for the study was the Melt Infiltrated SiC/SiC CMC system, which was initially developed under the Enabling Propulsion Materials Program (EPM) and is still under further refinement at NASA-Glenn Research Center (GRC). This material system has been systematically studied at various development periods and the most promising was the 01/01 Melt Infiltrated iBN SiC/SiC (01/01 is indicative of the month and year that development was frozen). There is a wide set of data from NASA for this system as well as a broad historic database from the material development. This allowed a testing system to be put into place to look for key development properties which would be needed from a modeling effort and would hence leverage existing data generated by NASA-GRC.

The Sylramic® fiber was fabricated by DuPont as a 10 μm diameter stochiometric SiC fiber and bundled into tows of 800 fibers each. The sizing applied was polyvinyl alcohol (PVA). For this study, the four lots of fibers, which were used, were wound on 19 different spools. The tow spools were then woven into a 5HS balanced weave at 20 EPI. An in-situ Boron Nitride (iBN) treatment was performed on the weave (at NASA-GRC), which created a fine layer of BN on every fiber. The fabric was then laid in graphite tooling to correspond to the final part design (flat plates for this experimental program). All the panels were manufactured from a symmetric cross ply laminate using a total of 8 plies. The graphite tooling has holes to allow the CVI deposition to occur. At this stage, another BN layer was applied. This BN coating was doped with Si to provide better environmental protection of the interface. This was followed by SiC vapor deposition around the tows. Typically, densification is done to about 30% open porosity. SiC particulates are then slurry cast into the material followed by melt infiltration of a Si alloy to arrive at a nearly full density material. The material at this time has less than 2% open porosity. Through this process, 15 panels were fabricated in 3 lots of material.

After fabrication, all the panels were interrogated by pulse echo ultrasound (10 MHz) and film X-ray. There was no indication of any delamination and no large scale porosity was noted in the panels. In addition, each panel had 2 tensile bars extracted for witness testing at room temperature. All samples tested failed above a 0.3% strain to failure requirement. Hence, all panels were accepted into the testing effort.

Tensile testing - Elastic modulus, shear modulus and Poisson's ratio at RT and 1204 ºC

All tensile testing was performed per EPM testing standards (equivalent ASTM C1359) with all of the witness testing done at room temperature (24ºC). The rest of the testing was done at 1204 ºC (2200ºF). Typical stress strain curves, shown in Figure 2, show a decrease in modulus, strength and strain to failure with temperature. The elastic modulus was calculated as the slope of the stress-strain curve in the linear region between 13.79 and 55.16 MPa (2-8 Ksi). The shear modulus was determined using biaxial extensometry on samples machined out of the cross ply laminate at 45°. The shear analysis was done consistent with ASTM D3518. The curves were fitted between 6.90 and 27.58 MPa (1- 4 Ksi) during the linear shear region to be consistent with the previous tensile modulus analysis. At room temperature, standard tensile bars were fitted with strain gages for the Poisson's ratio determination. Poisson's ratio was not determined at elevated temperatures.

Cyclic testing - RT and 1204 ºC

Cyclic tensile testing was performed on a series of samples at room temperature and 1204 ºC to evaluate the cyclic load response and damage progression under load. Typical stress-strain curves are shown in Figure 3 for room temperature testing and in Figure 4 for 1204 ºC. As can be seen, there is a slight decrease in the modulus, measured as the slope of the upload stress-strain curve between 13.79 and 55.16 MPa (2 and 8 Ksi) as the cycles progress indicating damage progression, as shown in Figure 5. A change in the slope of the unload segment of the stress-strain curve for RT, marked by an arrow in Figure 4, can also be observed as an indication of possible manufacturing compressive residual stresses of 45 ± 9 MPa similar to that observed in [4].

Strain at a given stress obtained from room temperature cyclic load experiments was subtracted from strain at the same stress for high temperature experiments to isolate the direct effect of temperature on the behavior. For each load cycle, marked by the peak stress of the hysteresis loop stress at unload, the strain was evaluated at unload stress of 27.58, 55.16 and 110.32 MPa (4, 8 and 16 Ksi). Strain values were plotted vs. the unload stress value for each peak stress unload value as shown in Figure 6. It can be seen that the difference in value of the strain continued to increase with the increase of the unload stress value indicating that mechanical damage progression that occurred at room temperature is accompanied by degradation of properties at higher temperatures. Such degradation may mainly be caused by oxidation and creep.

Through thickness testing - Mechanical and ultra-sound

One of the key elastic properties that is typically assumed and not measured is the through thickness modulus (Ez). Recent development efforts undertaken by NAS-GRC and the authors have arrived at a compressive test performed on a series of stacked disks to determine the through thickness modulus as a function of temperature. Each individual disk was ground flat so that there were no asperities. Enough disks were machined for a 2.54 cm extensometer to be flagged onto the sample. Through the center of each disk a hole was machined so that a graphite rod could be inserted to hold the stack in place and eliminate disk movement during initial loading. The rod was machined short so that it would not see any load that could affect the measured modulus. Even though the disks were machined, there was an initial compliance to the stack that had to be overcome by sufficient load. Once the linear region was reached, stress strain data was fitted to determine the modulus. The initial results from this effort are shown in Figure 7. As can be seen, the room temperature modulus was 72.4 GPa. An effort was undertaken by the Air Force Research Laboratory (AFRL) in conjunction with the University of Dayton Research Institute (UDRI) to determine this modulus by a through transmitted ultrasonic velocity measurement where the modulus is calculated as the density times the velocity squared, assuming an isotropic material. The ultrasonic value measured from the material taken from one of the panels for this program arrived at a value of 77.33 GPa in agreement with the mechanical methods. It should be noted that the stacked disk method samples a larger volume of material than the ultrasonic method.

Constituent Elastic Properties – Nano-indentation

Micro-structural characterization was done on samples from the three lots of materials and showed no microstructural differentiation which is consistent with mechanical property data. Nano-indentation work was conducted on the cross-sections of these samples to measure the elastic moduli of the discrete phases of the composite system [5]. This work was performed at room temperature using a NanoIndenter II at Oak Ridge National Laboratory (ORNL). The modulus was determined by analysis of the load-displacement recorded during nano-indentation as well as the value of the elastic modulus of the indentor [6]. The measured value of the elastic modulus of the Sylramic fiber was similar to that evaluated using tow test techniques [5].

For the BN phase, a Hysitron's TriboIndenter was used because it was capable of ultra low loads needed for the BN conducted at 400 μN. Additionally, the TriboIndenter allowed in-situ imaging by scanning probe microscopy as shown in Figure 8. The result of this work is listed in Table 1. It is important to mention that, up to the authors' knowledge, this is the first time that the Young's Modulus of the boron nitride was measured within a systematic procedure to evaluate the in-situ elastic moduli of constituent phases.

Analytical modeling

Analytical models used to predict the elastic properties of textile composite materials are available in literature (e.g., [7-9]) varying from closed form to numerical solutions. Most of these models lack the detailed geometry that can properly represent the composites internal fabric structure which may have a strong impact on the mechanical behavior of the textile composites with ceramic or polymer matrices. For example, 2D fabrics offer in-plane characteristics that are superior to those of 3D fabrics, while the opposite is true for out-of-plane properties. Quantification of these properties requires an understanding of the spatial location of yarns and the contribution of each composite constituent to the composite overall response. To this end, a geometric model was developed based on fabric processing to evaluate the spatial location of yarns [10]. The knowledge of yarn location and matrix distribution provide basis for possible application of numerical mechanical models, such as traditional finite element analysis (FEA). Unfortunately, the use of traditional FEA is limited by the complexity of the preform geometry and meshing problems, and requires a large number of elements. A typical plain weave fabric would require a few thousands elements to model [11]. It is expected that other complex fabrics, such as 3D preforms, would require hundreds of thousands of elements.

To solve this problem a hybrid FEA was developed [3] where a unit cell of the fabric preform is identified and divided into hexahedra brick elements with fiber and matrix around each integration point. Material homogenization was carried out around each integration point to define the anisotropic material response [12]. The boundary conditions of the unit cell dictated by the assumption of repeatability and continuity helped reduce the size of the unit cell stiffness matrix. A virtual work technique was used to calculate the elastic properties of the unit cell. Both geometric and mechanical FEA models were combined and integrated using visual C++ into a computer algorithm called “pcGINA”.

Comparison of analytical modeling to experimental data

Material properties of constituents are listed in Table 1 along with their relative volume fractions used in the analysis. The moduli were obtained from nano-indentation experiments mentioned above. The Poisson’s ratios of constituents were obtained from [13] and the shear moduli were calculated using elasticity equations assuming that all constituent phases are locally isotropic. An approximation was carried out by dividing the composite into two parts – coated fiber comprised of iBN-Sylramic® fibers coated with Si-doped BN and a matrix formed from CVI-SiC, SC-SiC and Si. Properties of the coated fiber were calculated based on the micromechanics model developed in [14]. The micrographic image shown in Figure 1 revealed a shiny material (Si) mixed with another grayish color material (SC-SiC) in the place between the yarns with some dark areas that are most probably voids. Based on this micrograph an in-series model was used to calculate the combined properties of SC-SiC and Si (iso-stress model) and an in-parallel model (iso-strain model) was used to combine these properties with the properties of CVI-SiC. Utilizing this approach the matrix properties were calculated as E_m= 329.29 GPa, G_m = 139.08 GPa, and ν_m=0.182.

Calculation of room temperature elastic properties using pcGINA was compared to experimental data as shown in Table 2 using 225 eight-noded hexahedra brick elements shown in Figure 9. Table 2 includes the results for composites with and without voids showing a range of results for the effect of voids. This range is due to the lack of information on the exact location of voids. Typically, the upper bound values are for the case when voids exist in a spherical form in the matrix away from the fibers, while the lower bound values are when voids that are aligned within yarns and at yarn cross-over points. It can be seen from the table that estimates for in-plane tensile moduli (E_x and E_y), shear modulus (G_{xy}) and Poisson's ratio (ν_{xy}) are very close to the experimental data.

The value for the through-thickness modulus (E_z) calculated by pcGINA is approximately three times higher than the experimental value reported in Table 2. But it was observed that the experimental value of E_z is lower than the moduli of major composite constituents listed in Table 1 which prompted further investigation. Micrographic images of yarns showed intra-laminar voids between fibers of approximately 10% of the yarn cross-section area. An example from these images is shown in Figure 10. Investigation of the effect of compressive load level on the value of E_z for the composite under investigation showed dependence of modulus value on the load level as shown in Figure 11. The modulus showed abrupt increase at low load levels and was almost flat (i.e., gave constant value) of 138 GPa at stresses higher than 300 MPa. It is possible that the existence of voids caused the reduction of modulus at low load levels, but as the compressive load increased the voids collapsed causing an increase in the value of the through thickness modulus.

As a further validation of the stacked disk experimental approach, a rod of MACOR machineable glass ceramic was acquired as a monolithic material. A 28 mm long piece of the rod was tested in compression to obtain the elastic modulus which was determined to be 68.9 GPa. In addition, a series of disks were machined out of the rod with the same dimensions as that used in the testing done on the CMC material. Flags were attached to the specimen and the testing was conducted at room temperature in the same controlled manner as the CMC material. By this method, the modulus of the material was determined to be 66.0 GPa. The literature value for the elastic modulus is 66.9 GPa. The stacked method showed the same compliance issues under low stress levels observed with the CMC material as shown in Figure 11. This strong agreement between testing of monolithic material and stacked disks shows that the stacked disk method can be accepted as a sound testing method for composite material.

Data available in literature on the effect of temperature on various composite constituents was used to evaluate the change of elastic properties of the composite from room temperature to 1204 ºC. As expected, it was much easier to find data on Si metal than on α and β silicon carbides or Si doped BN. Elastic constants of silicon were reported to depreciate with temperature in a linear fashion up to around 1220 ºC [15, 16]. The elastic moduli of covalent carbides were reported in the National Institute of Standards and Technology (NIST) structural ceramics database [17] to also follow a linear depreciation pattern. Using data from these sources the properties of the matrix at 1204 ºC were calculated as E_m= 302.84 GPa, G_m = 127.90 GPa, and ν_m=0.182. Data for the change of properties of Si doped BN with temperature was not available in literature, and it was assumed that the BN coat properties will not change. The reduction of mechanical properties of Sylramic® fibers with temperature was also not available according to personal communications with the manufacturer. Change of properties of other SiC fibers such as Hi-Nicalon and CG Nicalon are available [18] and was similar to values of covalent SiC [17]. This data was used in the current analysis for Sylramic® fiber. Table 3 shows a comparison between experimental data and modeling values. It can be seen that results of pcGINA runs show reasonable estimates for the value of the composite in-plane tensile and shear moduli depreciation with temperature. No experimental data was available for Poisson's ratio.

Conclusions

Fifteen panels were manufactured and coupons were taken to evaluate the room temperature and high temperature elastic properties of SiC/SiC composites (01/01 material). Elastic modulus and shear modulus were evaluated at both temperatures and showed depreciation of properties with temperature. Other experiments were conducted to evaluate the through thickness modulus and the in-plane Poisson ratio. Cyclic load experiments were conducted at room temperature and 1204 ºC to quantify the change of elastic modulus with temperature and with damage progression. The effect of temperature on the depreciation of properties under cyclic loading was isolated by subtracting high temperature strain values from strain values at room temperature. Nano-indentation experiments were conducted to evaluate the in-situ moduli of constituent materials at room temperature. A micromechanics model based on hybrid FEA for a unit cell was used to model the composite response. Properties of constituent materials at room temperature reported from nano-indentation experiments and 1204 ºC found in literature were used in the model. Close agreement was observed with elastic and shear moduli at room temperature and 1204 ºC and Poisson's ratio at room temperature.

Experimental value for the through thickness modulus utilizing the stacked-disk testing approach was lower than the calculated value and also lower than moduli of major composite constituents. Micrographic images showed voids between fibers inside yarns and further experimental investigation of the stacked-disk testing method showed a dependence of the value of through-thickness modulus, for the CMC material and a monolithic glass ceramic, on the level of compressive stress at low stress levels, possibly due to surface asperities.

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Table 1: Properties of constituent material at room temperature (GPa):

Property	Sylramic® fiber (β-SiC)	BN Coating (Si-doped BN)	SiC-CVI (β-SiC)	SiC-MI		Porosity
				α-SiC	Si	
E^*	394.95	20.27	438.80	405.63	164.86	
$G^@$	168.79	8.27	187.54	173.34	67.57	
$\nu^\#$	0.17	0.22	0.17	0.17	0.22	
Vf	0.36	0.072	0.23	0.177	0.135	0.026

* Nano-indentation
# [13]
@ Calculated

Table 2: Experimental and analytical modeling of elastic properties at room temperature (GPa):

Property	Without voids	With 2.7% voids		Experiment
		Lower bound	Upper bound	
E_x, E_y	269.46	224.29	263.04	274.83 ± 10.34
E_z	213.75	202.99	208.99	72.40 ± 6.21
Gxy	92.39	88.46	90.39	95.84
G_{xz}, G_{yz}	90.12	86.46	87.77	---
ν_{xy}	0.14	0.161	0.146	0.127 ± 0.003
ν_{xz}, ν_{yz}	0.195	0.188	0.204	---

Table 3: Experimental and analytical modeling of elastic properties at 1204 ºC (GPa):

Property	Without voids	With 2.7% voids		Experiment
		Lower bound	Upper bound	
E_x, E_y	246.57	204.37	246.15	233.74 ± 19.51
E_z	200.64	190.58	200.37	60.47 ± 0.69
Gxy	86.60	82.88	86.53	84.12
G_{xz}, G_{yz}	84.67	81.22	84.60	---
ν_{xy}	0.144	0.166	0.144	---
ν_{xz}, ν_{yz}	0.193	0.186	0.193	---

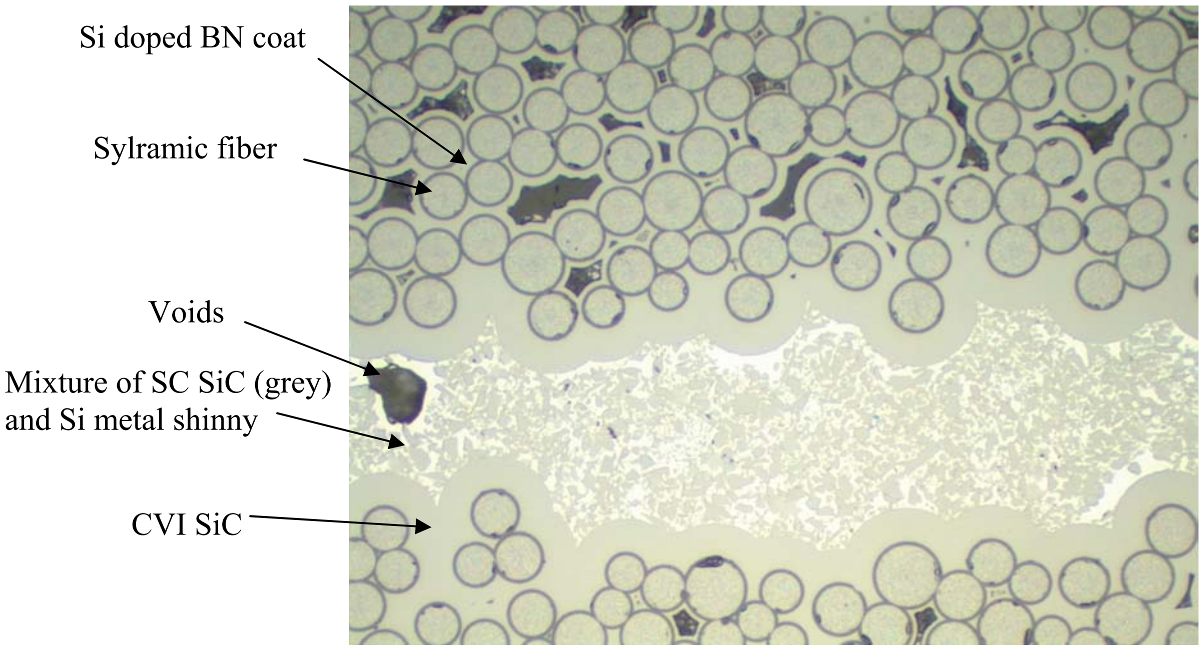


Figure 1: Micrographic image of 5HS SiC/SiC composites

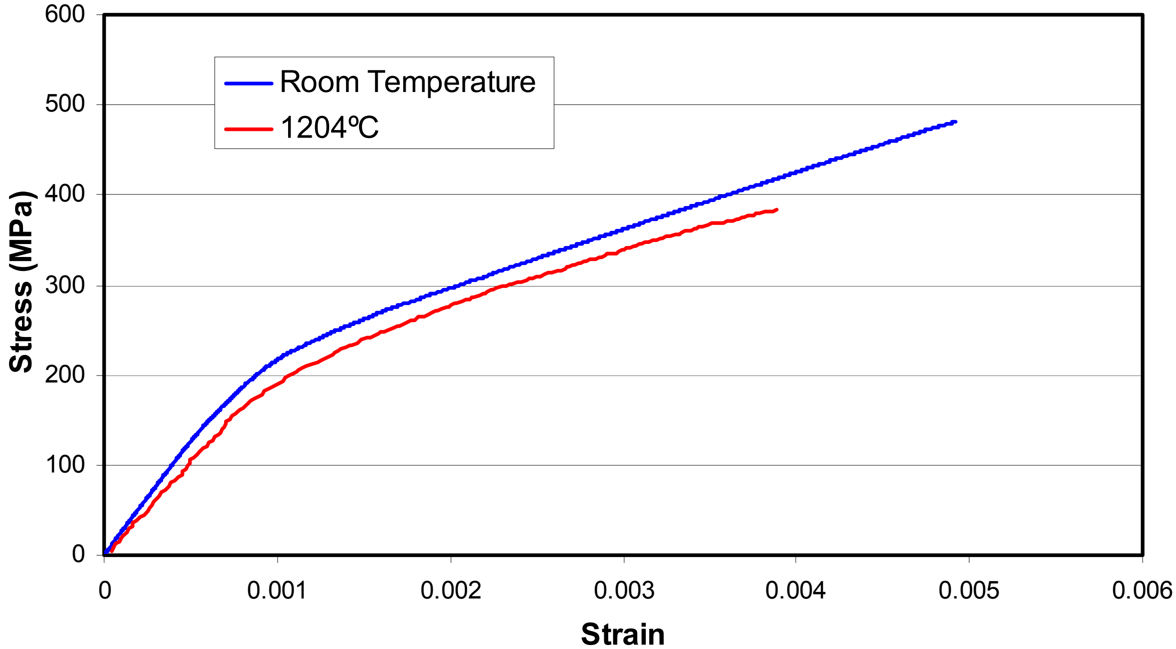


Figure 2: Tensile stress-strain curves at room temperature and 1204 ºC

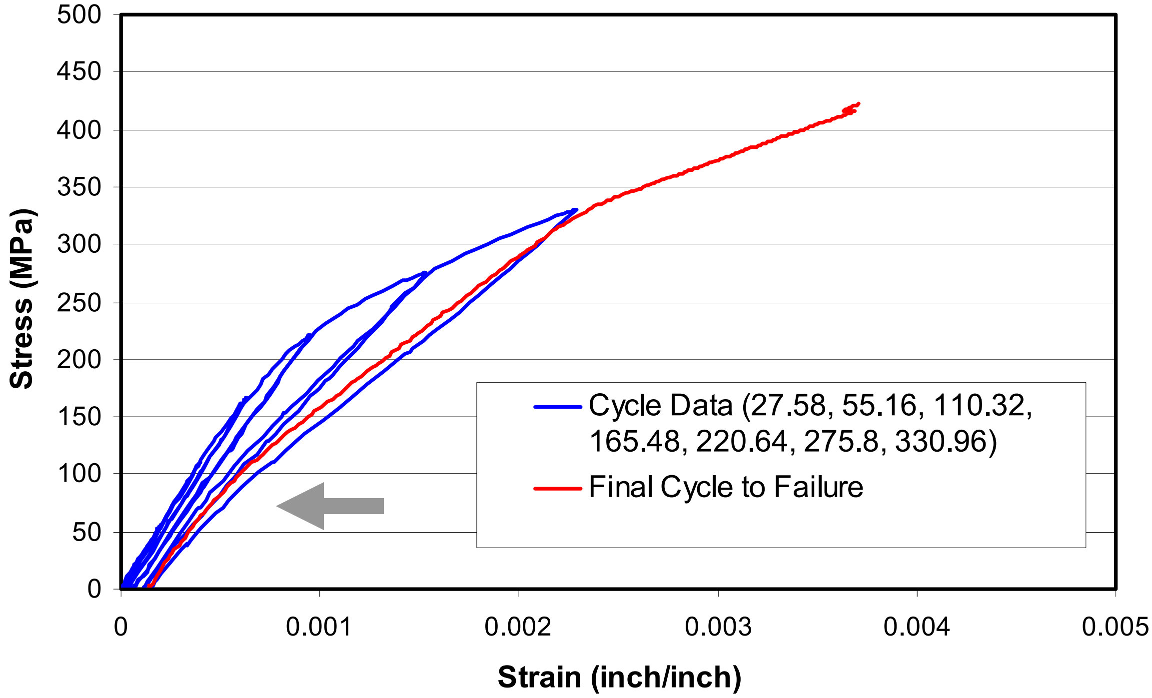


Figure 3: Cyclic stress-strain curve at room temperature. . Arrow is pointing at area of change in the slope of the unload segment of the curve.

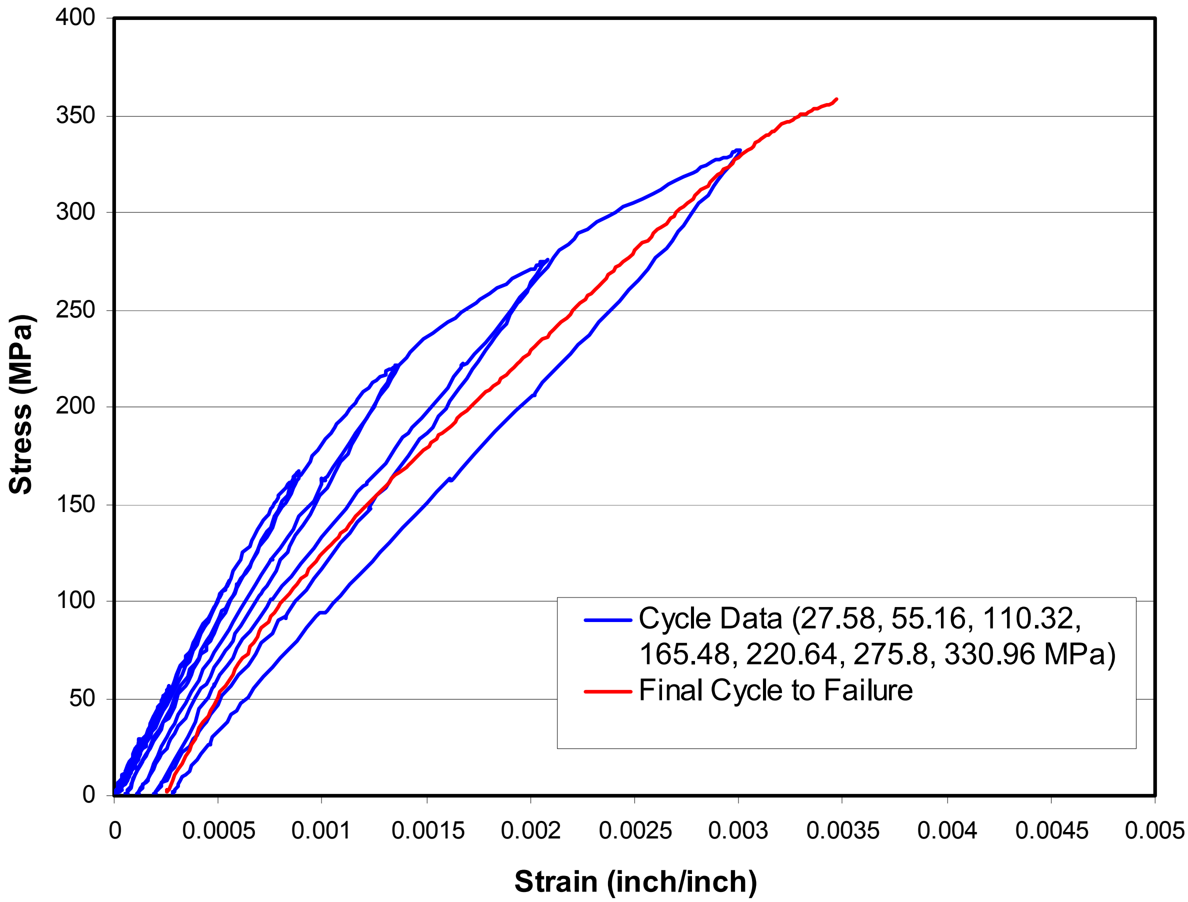


Figure 4: Cyclic stress-strain curve at 1204 ºC. Arrow is pointing at area of change in the slope of the unload segment of the curve.

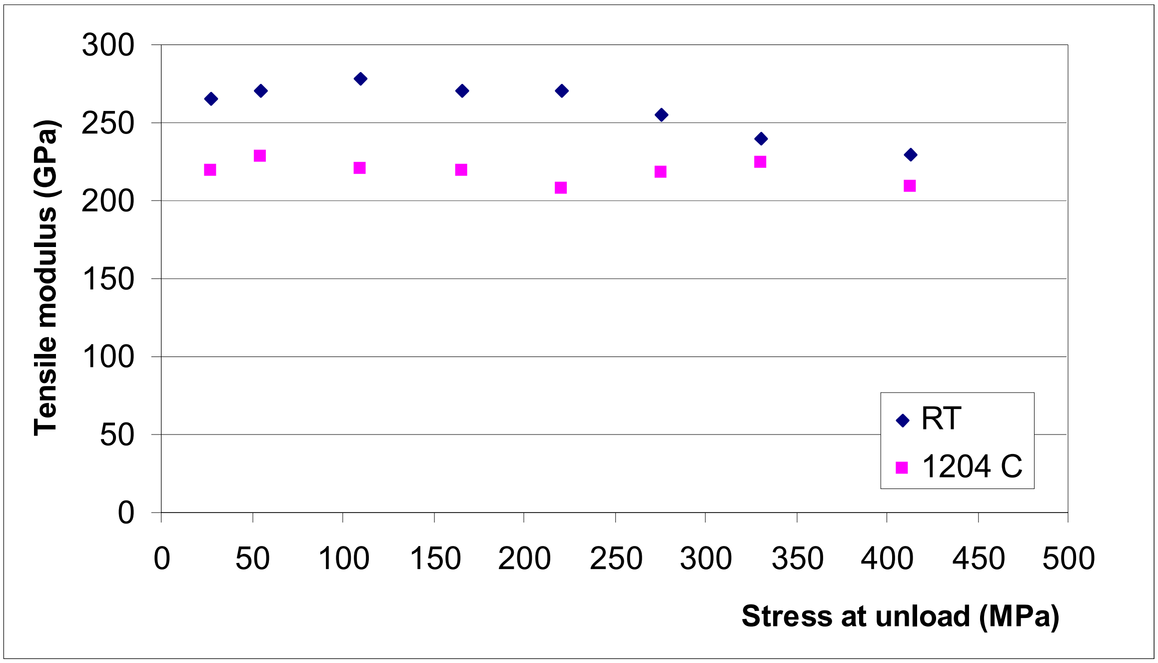


Figure 5: Effect of cyclic load and temperature on modulus

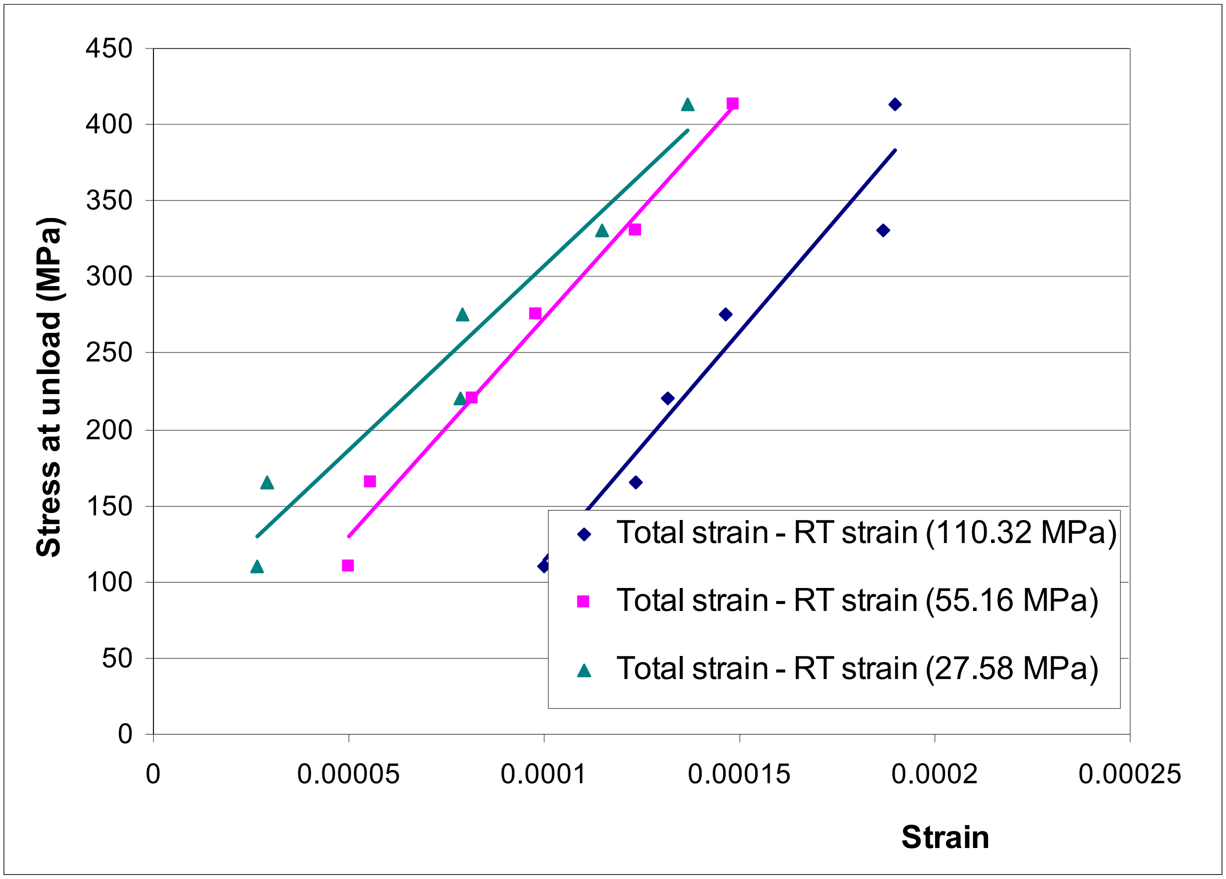


Figure 6: Total deformation at 1204 ºC minus mechanical deformation at room temperature under cyclic load for 27.58, 55.16, and 110.32 MPa

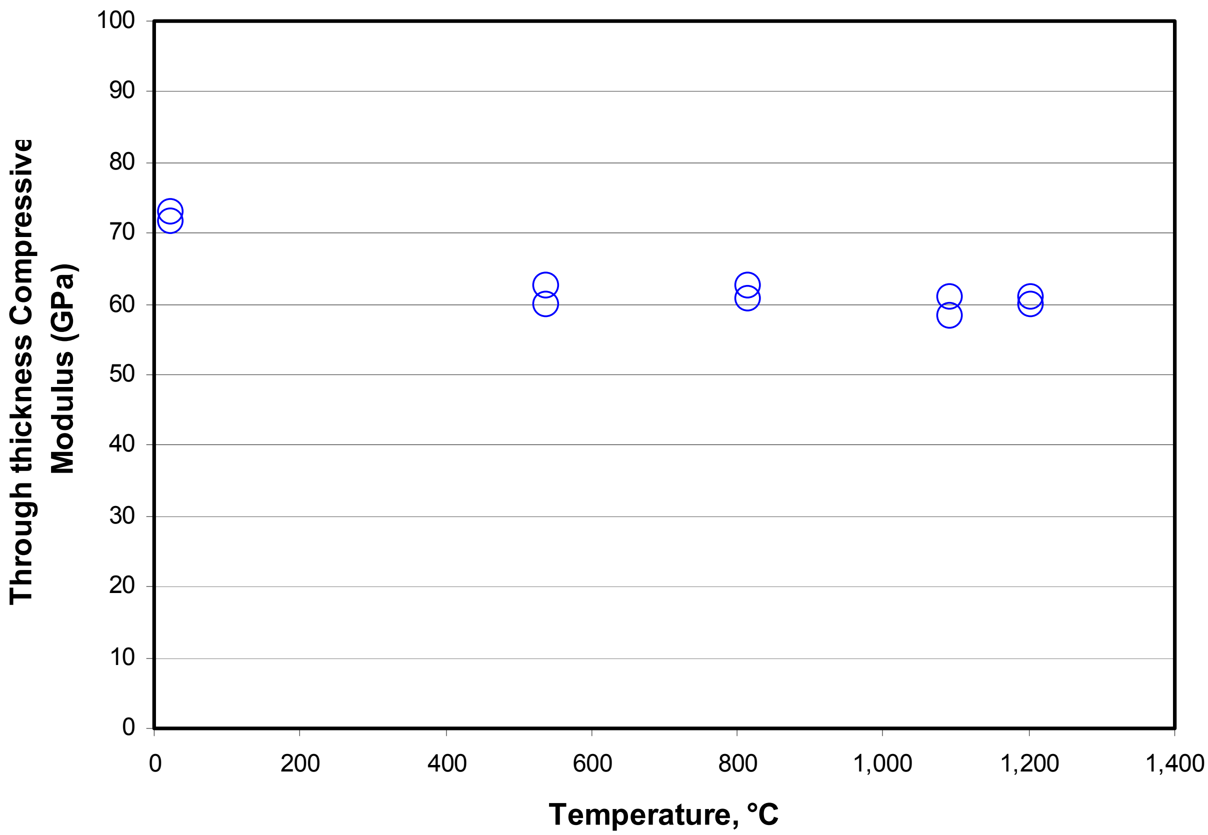
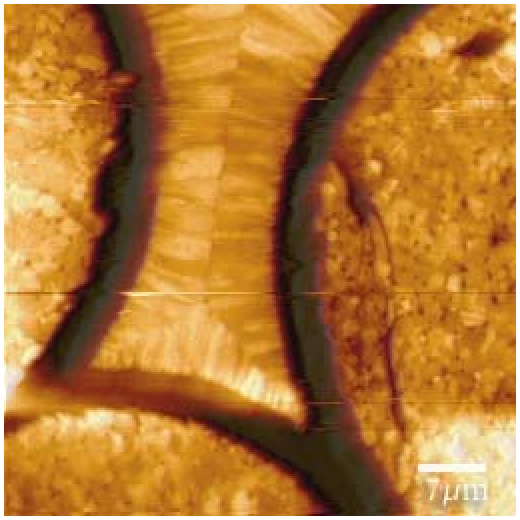
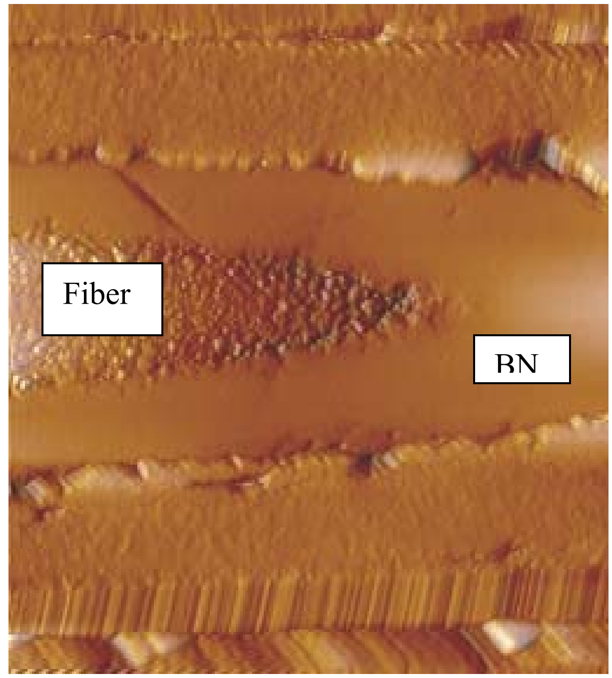


Figure 7: Through the thickness modulus (Ez) versus temperature



a) CVI SiC between 3 SiC fibers



(15 µm x 15 µm scan)

b) BN around fiber (longitudinal cross section). Fiber is cut at an oblique angle

Figure 8. Scanning probe microscopy of CVI SiC and BN near SiC Fibers.

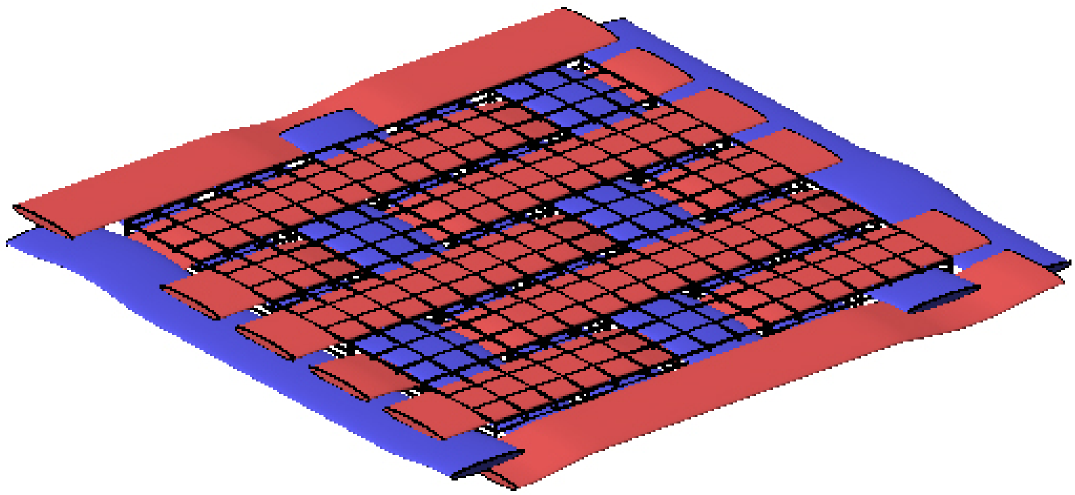


Figure 9: Geometric model and FE mesh of 5-harness satin woven SiC/SiC composite

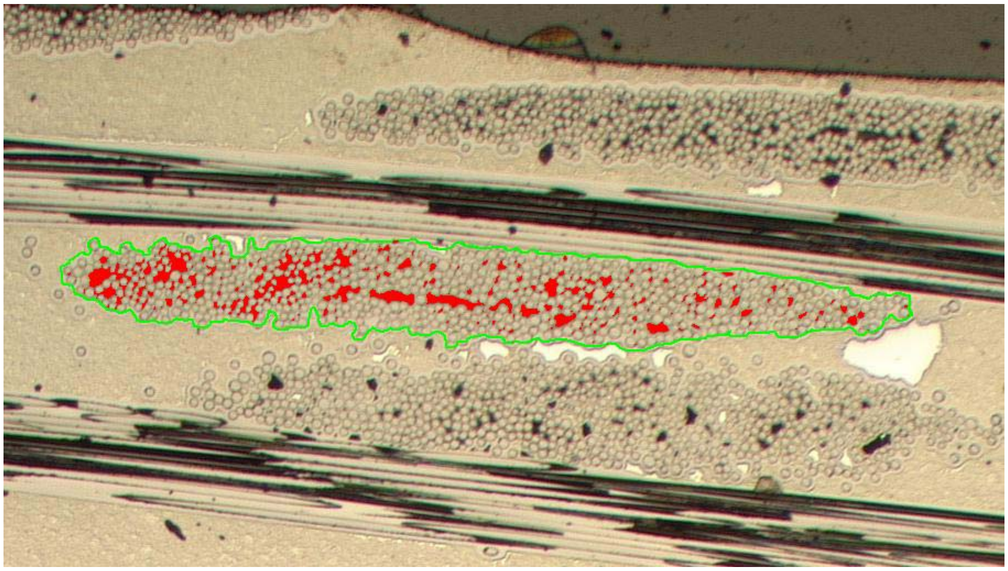


Figure 10: Micrographic images showing voids between fibers inside the yarn

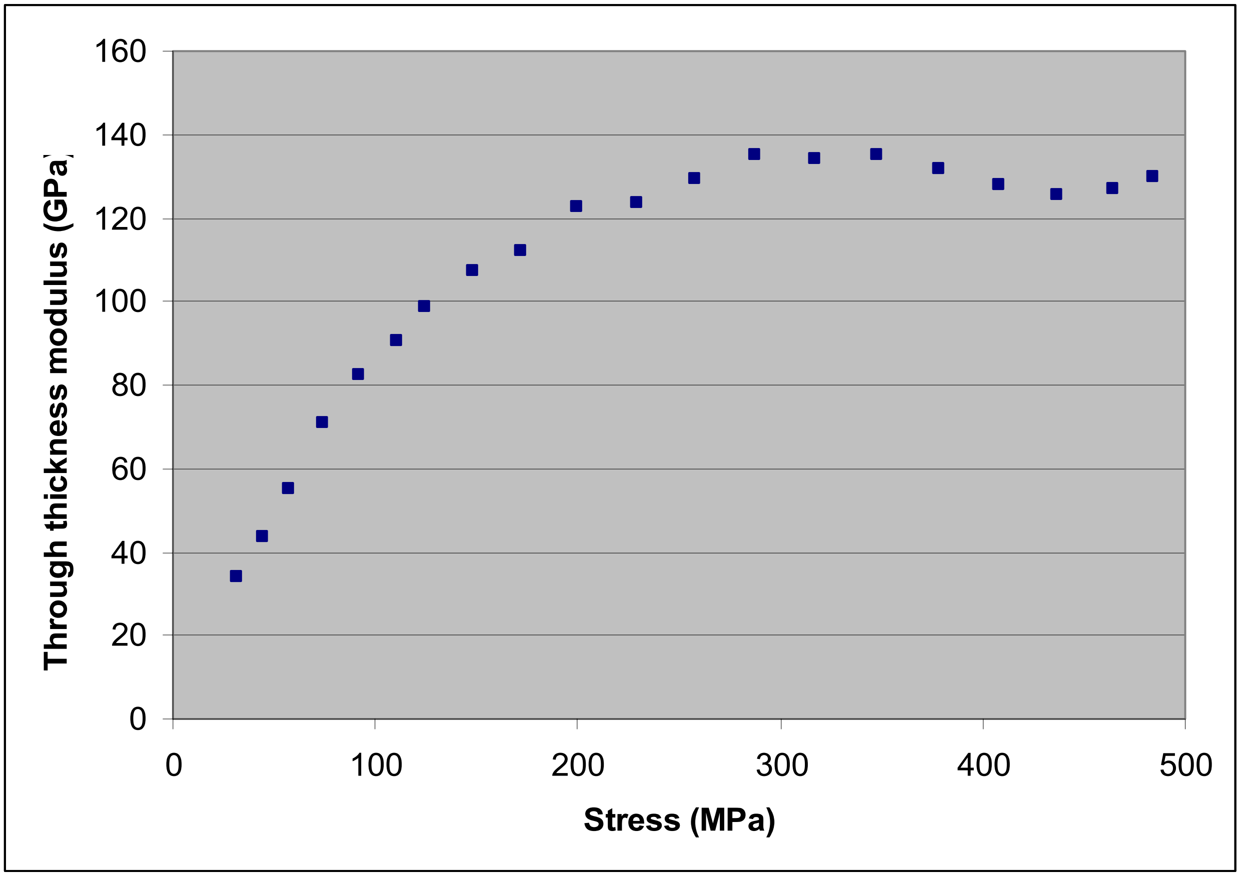


Figure 11: The effect of load level on the through thickness elastic modulus of SiC/SiC composites